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Influence of Thermal, Hydrothermal, and Acid–Base Treatments on Structural Stability and Surface Properties of Macro-, Meso-, and Microporous Carbons

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Effects of thermal treatment (at 723 K), hydrothermal treatment [low-pressure steam vapor (34 Torr) at 723 and 923 K and water (at autogenous pressure) at 398 and 473 K], and acid–base treatment (at 473 K) on the structural stability and physical properties of porous carbon samples prepared from both natural and synthetic sources have been studied. These thermally, hydrothermally, and acid–base-treated carbons have been characterized using BET, thermogravimetric, and infrared techniques. Investigations of the treated carbon samples revealed that the thermal stability, pore structure, pore size distribution, and surface area are strongly affected by the thermal, hydrothermal, and acid–base treatments. The method of sample treatment influences the order of thermal stability and porosity and surface area (differences in surface area were attributed to differences in porosity, based on micropore and total pore volume). FTIR studies show that the carbon structures are significantly influenced by the hydrothermal and acid–base treatments. The surface and pore structure modifications of the microporous carbon have also been studied by controlled air decomposition and high temperature aliphatic and aromatic organic vapor deposition. **© 1999 Academic Press**

Key Words: **macro-, meso-, and microporous carbons; surface area; pore size distribution; controlled decomposition; FTIR; thermal, hydrothermal, acid–base treatment; TGA; BET.**

INTRODUCTION

In many adsorption–separation processes, with both laboratory and industrial scale applications, activated carbons are widely used as adsorbents for the removal of organics\gas purifications and as a support material in various catalytic processes (1–5). The macro-, meso-, and microporous carbons are of considerable interest as adsorbents for the removal of bulkier organics from various industrial processes (6–8). The activated carbons are of great research interest because of their high surface area, chemical inertness, and ability to endure harsh chemical environments.

In our earlier studies (9), the preparation and characterization of heat-treated carbons obtained from natural and synthetic (artificial) sources and the ability of these carbons to remove Bayer active organic compounds from simple aqueous systems were investigated. In various adsorption processes, the functioning of an activated carbon material is strongly dependent on the factors such as surface area, macro-, meso-, and mircopore size/volume, pore size distribution, and structure. Thus, from the point of view of activated carbon application as an adsorbent and/or catalyst support, knowledge of the influence of various thermal, hydrothermal, and acid–base treatment on its surface and structural stability is of great interest. In the present study, the influence of thermal, hydrothermal, and acid–base treatments on the thermal stability, surface area, pore size distribution, and structure of activated carbons are investigated. Effects of the controlled decomposition and high temperature organic vapors on the surface area and pore size distribution of the microporous carbon have also been studied.

EXPERIMENTAL

Preparation of Carbon Samples

Heat-treated carbons were prepared from pure (100%) mustard oil (C1), almond oil (C2), and olive oil (C3). In each case, the heat-treated carbon sample was prepared by controlled burning of the source at 900–1123 K for 5–6 h. The carbon sample C4 was obtained commercially. The procedures used for the thermal, hydrothermal, and acid–base treatment of carbon samples are as follows.

Thermal Treatment

Microporous carbon (C4) (4 g) was calcined at 723 K for 7 h (in the presence helium) in a stainless steel tube reactor furnace.

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TABLE 1
Source and Treatment of the Carbon Samples

Sample code	Source	Treatment type
C1	Mustard oil	Fresh
C1–H1	Mustard oil	Hydrothermal treatment at 398 K
C1–H2	Mustard oil	Hydrothermal treatment at 473 K
C1–A	Mustard oil	Acidic hydrothermal treatment at 473 K
C1–B	Mustard oil	Basic hydrothermal treatment at 473 K
C2	Almond oil	Fresh
C2–H1	Almond oil	Hydrothermal treatment at 398 K
C2–H2	Almond oil	Hydrothermal treatment at 473 K
C2–A	Almond oil	Acidic hydrothermal treatment at 473 K
C2–B	Almond oil	Basic hydrothermal treatment at 473 K
C3	Olive oil	Fresh
C3–H1	Olive oil	Hydrothermal treatment at 398 K
C3–H2	Olive oil	Hydrothermal treatment at 473 K
C4	Commercial high surface area carbon	Fresh
C4–H1	Commercial high surface area carbon	Hydrothermal treatment at 398 K
C4–H2	Commercial high surface area carbon	Hydrothermal treatment at 473 K
C4–A	Commercial high surface area carbon	Acidic hydrothermal treatment at 473 K
C4–B	Commercial high surface area carbon	Basic hydrothermal treatment at 473 K
C4–C	Commercial high surface area carbon	723 K He 7 h
C4–D	Commercial high surface area carbon	723 K Steam 7 h
C4–E	Commercial high surface area carbon	923 K Steam 7 h
C4–F	Commercial high surface area carbon	673 K, Controlled air decomposition
C4–G	Commercial high surface area carbon	773 K, Controlled air decomposition
C4–H	Commercial high surface area carbon	673 K, *n*-Hexane 10 pulses of 10 μl
C4–I	Commercial high surface area carbon	673 K, *n*-Hexane 10 pulses of 100 μl
C4–J	Commercial high surface area carbon	673 K, Toluene 10 pulses of 10 μl
C4–K	Commercial high surface area carbon	773 K, Toluene 10 pulses of 10 μl

Hydrothermal Treatment

Low-pressure steam treatment. A mixture of water vapor (partial pressure, 34 Torr) and helium at a flow rate of 120 $cm^3 min^{-1}$ was passed through a bed of carbon at 723 and 923 K for 7 h.

Water treatment. Activated carbon (4 g) was immersed in water and heated in a pressure bomb at 473 K for 5 h at autogenous pressure. After treatment, the carbon sample was filtered and dried in a vacuum oven at 373 K for 16 h.

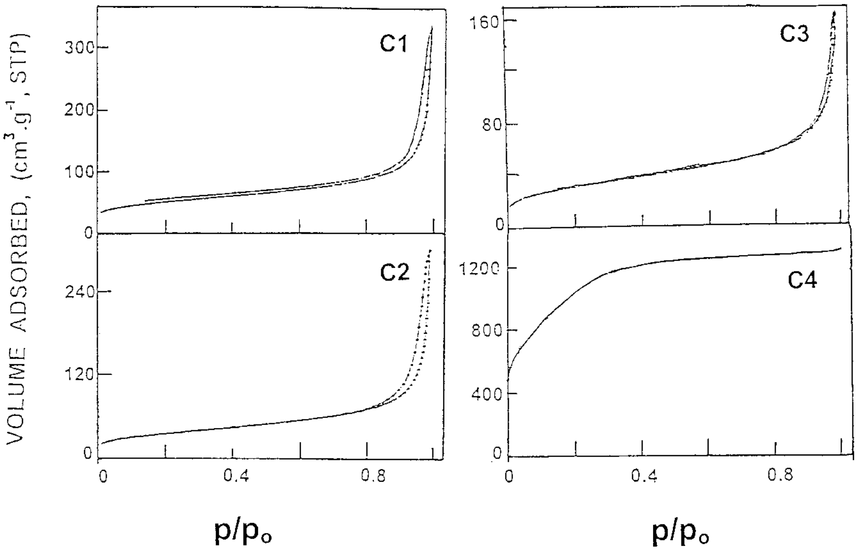

FIG. 1. Nitrogen adsorption/desorption isotherms for fresh carbon samples obtained from different sources.

TABLE 2
Surface Properties of the Carbon Samples

Sample	BET surface area ($m^2\ g^{-1}$)	External surface area ($m^2\ g^{-1}$)	Micropore surface area ($m^2\ g^{-1}$)	Micropore volume ($cm^3\ g^{-1}$)	Single point total pore volume of pores < [Å] ($cm^3\ g^{-1}$)	Average pore diameter (4V/A by BET) (Å)
C1	174	126	48	0.022	0.36 [1100 Å]	82.6
C1–H1	132	132			0.30 [1251 Å]	90.5
C1–H2	90				0.29 [1146 Å]	126
C1–A	139	121	18	0.007	0.44 [870 Å]	128
C1–B	101	101			0.39 [1222 Å]	167
C2	108	89	19	0.008	0.20 [1157 Å]	74.3
C2–H1	74	74			0.17 [13560 Å]	92
C2–H2	67	67			0.20 [1298 Å]	117
C2–A	94	90	4	0.003	0.37 [1011 Å]	111
C2–B	102	94	8	0.005	0.41 [931 Å]	117
C3	123	103	20	0.009	0.34 [1114 Å]	109.4
C3–H1	92				0.30 [1165 Å]	133
C3–H2	69	69			0.23 [1320 Å]	165
C4	3900		498	0.135	1.9 [2307 Å]	20.75
C4–H1	2645				1.6 [2174 Å]	21.9
C4–H2	2178				1.14 [2084 Å]	22
C4–A	1344				0.68 [3713 Å]	14.9
C4–B	2547				1.3 [1543 Å]	21.6
C4–C	3890				2.03 [2427 Å]	20.8
C4–D	3885				2.066 [2172 Å]	21
C4–E	3781				1.92 [1331 Å]	20.3
C4–F	3810		431	0.10	1.81 [1714 Å]	20.5
C4–G	3790		452	0.11	2.03 [5588 Å]	20.4
C4–H	3397		332	0.065	1.73 [2188 Å]	20.4
C4–I	3432		307	0.055	1.75 [3579 Å]	20.45
C4–J	3411		492	0.136	1.72 [1487 Å]	20.14
C4–K	3405		474	0.131	1.728 [2880 Å]	20.3

Acid–base treatment. Activated carbon (4 g) was immersed in 4 M acid (HCl)/base (NaOH) solution (200 cm^3) and heated in a pressure bomb at 473 K for 5 h at autogenous pressure. After treatment, the carbon sample was filtered and dried in a vacuum oven at 373 K for 16h.

Controlled air decomposition. The controlled decomposition of microporous carbon (C4) was carried out by injecting 30 air pulses (each 200 μl) after every 15 min in the presence of helium (flow rate of 80 $cm^3\ min^{-1}$) at 673 and 773 K for 7 h. The carbon (500 mg) was packed in an stainless steel tube reactor placed in a split furnace. Before the controlled air decomposition, the carbon was heated at 673 K for 1 h in a flow of helium.

High temperature organic deposition. The controlled organic deposition of microporous carbon (C4) was carried out by injecting 10 *n*-hexane/toluene pulses of 10/100 μl (each 15 min) in the presence of helium (flow rate of 40 $cm^3\ min^{-1}$) at 673/773 K. The carbon (500 mg) was packed in an stainless steel tube reactor placed in a split furnace. Before the hydrocarbon deposition, the carbon was heated at 673 K for 1 h in a flow of helium.

Characterization

The following methods were used for characterising the carbon samples.

1. BET: The BET surface area, pore volume and nitrogen adsorption/desorption isotherms were obtained using a Micromeritics ASAP2000 surface area analyzer. Pore volume and pore size distribution were determined using the Barrett, Joyner, and Halenda (BJH) method (10). Each carbon sample was degassed at 473 K under vacuum for a minimum of 24 h.
2. Thermogravimetric: Thermal analysis of the carbon samples, dried at 373 K in air for 24 h, was performed under inert and oxidizing atmospheres, using a fully automated, computer controlled Perkin Elmer TGA7 thermal analyzer. Thermal analysis was performed using a sample mass of 5–6 mg and a platinum crucible sample holder. The temperature range employed was 303–1173 K, with a heating rate of 5 K min^{-1} and an atmosphere of nitrogen or air (flow rate, 100 $cm^3\ min^{-1}$).

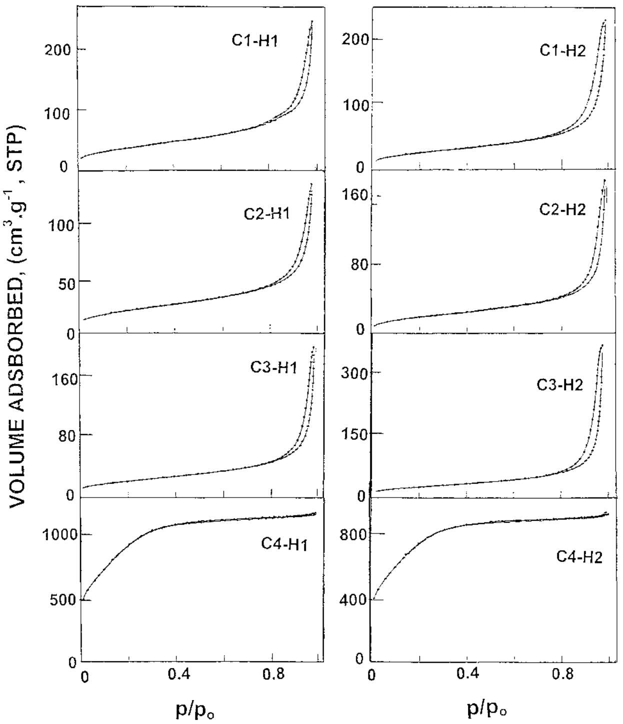

FIG. 2. Nitrogen adsorption/desorption isotherms for the hydrothermally modified carbon samples at 398 and 473 K.

3. Infrared spectroscopy (IR): FT-infrared spectra of the samples in the lattice vibration region were obtained from a KBr disk (1.2 wt%) at 2 cm^{-1} resolution using a Perkin Elmer FT-IR 1725X spectrometer.

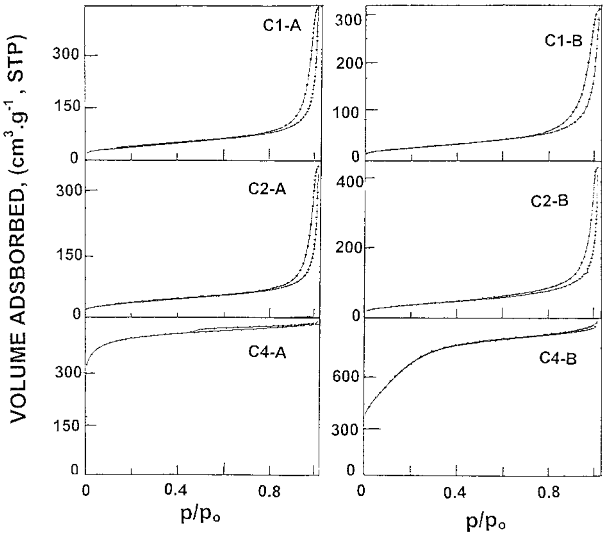

FIG. 3. Nitrogen adsorption/desorption isotherms for the hydrothermally modified carbon samples in acidic and basic medium at 473 K.

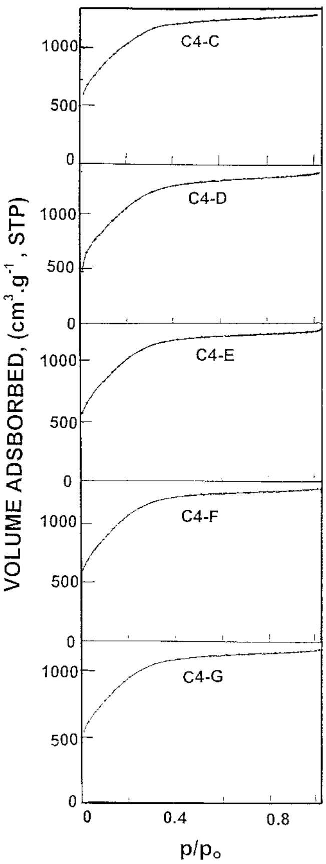

FIG. 4. Nitrogen adsorption/desorption isotherms for the carbon samples modified by high-temperature treatment (C4–C), high-temperature steam treatments [(C4–D at 723 K) (C4–E at 823 K)], and controlled air decomposition at 673 K (C4–F) and 773 K (C4–G).

RESULTS AND DISCUSSION

The carbon samples obtained from different sources along with their sample code and type of treatment are presented in Table 1. In our previous studies (9), the carbon, hydrogen, and nitrogen contents of the carbon samples obtained from mustard oil, almond oil, and olive oil and other sources were reported. The carbon, hydrogen, and nitrogen contents of the carbon samples from vary from sample to sample.

Figure 1 shows the nitrogen adsorption–desorption isotherms for the fresh carbon samples obtained from mustard oil (C1), almond oil (C2), and olive oil (C3) and highly microporous carbon (C4). The BET surface area, external and microsurface area, micropore volume, and total pore volume for the carbons investigated are listed in Table 2. Nitrogen adsorption at 77 K by the carbons prepared from mustard oil (C1), almond oil (C2), and olive oil (C3) leads to type IV isotherms (Fig. 1) (11). The isotherms for the C4 carbon (Fig. 1) show very high nitrogen uptakes in compar-

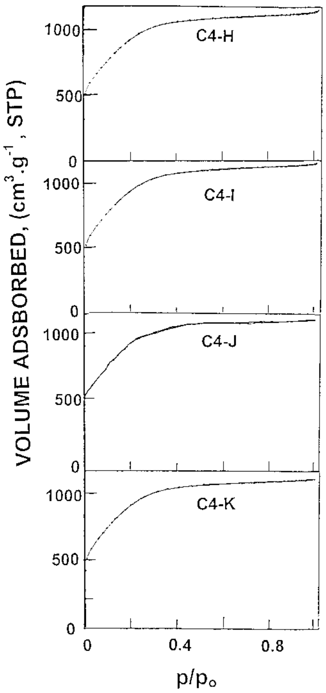

FIG. 5. Nitrogen adsorption/desorption isotherms for the carbon samples modified by high-temperature aliphatic and aromatic hydrocarbon treatments.

ison to other carbon samples, indicating the presence of a high level of porosity.

The nitrogen adsorption–desorption isotherms for the hydrothermally modified carbon samples at 398 and 473 K are shown in Fig. 2. The BET surface area, external and micro-surface area, micropore volume, and total pore volume for the carbons investigated are listed in Table 2. The BET results show that the hydrothermal treatment affects the surface area (external and micropore) and pore volume (macro- and micropore) of the carbon samples. At the hydrothermal treatment of 398 K, the surface area of the carbons (C1–C4) was decreased by 24 to 31%. The data (Table 2) indicated that the increase in the hydrothermal temperature of carbons from 398 to 473 K results in a significant decrease in their surface area by 38 to 48%. The hydrothermal treatment of carbon sample causes partial degradation of the macro- and micropore structures, thus affecting the surface area and pore volume.

For studying the effect of the high-temperature acid–base treatment, the carbons prepared from natural (mustard oil and almond oil) and synthetic sources were used. The nitrogen adsorption–desorption isotherms for the acid–base-treated carbon samples (Fig. 3) are different than those of the fresh carbon samples. In the nitrogen adsorption–desorption isotherms, the major changes are observed in the hysteresis loop. After the acid–base treatment, the width of hysteresis loop is increased, indicating the formation of more macropores. The acid–base treatment reduces the surface area and pore volume of carbon samples (Table 2). For the carbon sample (C1) prepared from the mustard oil, the influence of base treatment is more pronounced on the surface area than the acid treatment, while the reverse is observed for the carbon samples (C2, C4). The acid treatment of the microporous carbon (C4) (pre-

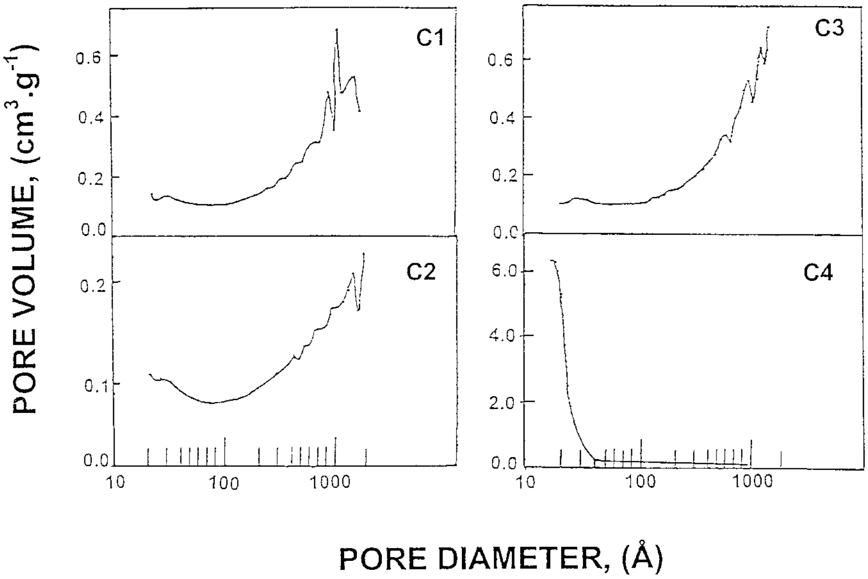

FIG. 6. The pore volume distributions (adsorption) for fresh carbon samples obtained from different sources.

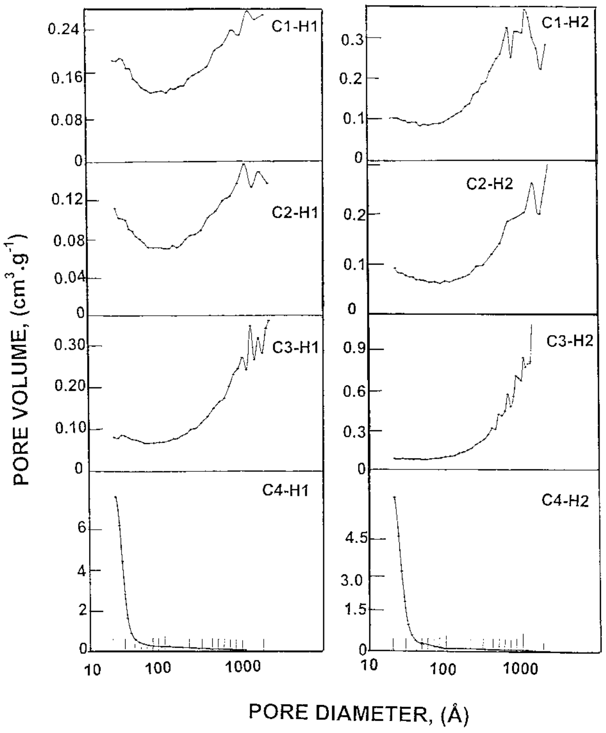

FIG. 7. The pore volume distributions (adsorption) for the hydrothermally modified carbon samples at 398 and 473 K.

pared by synthetic source) decreases the surface area by more than 65%.

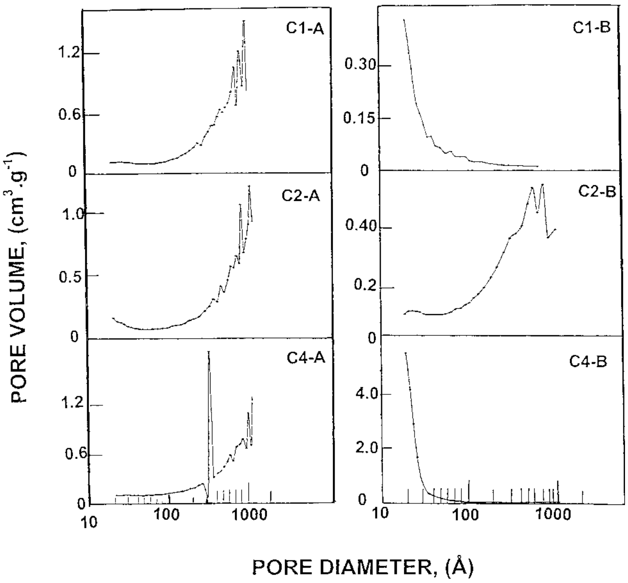

FIG. 8. The pore volume distributions (adsorption) for the hydrothermally modified carbon samples in acidic and basic medium at 473 K.

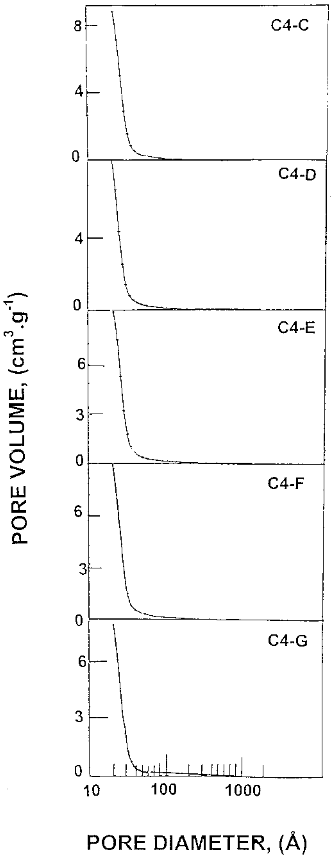

FIG. 9. The pore volume distributions (adsorption) for the carbon samples modified by high-temperature treatment (C4–C), high-temperature steam treatments [(C4–D at 723 K) (C4–E at 823 K)], and controlled air decomposition at 673 K (C4–F) and 773 K (C4–G).

The microporous carbon (C4) (high surface area) was used to investigate the effect of high temperature, high temperature steam, and controlled air decomposition. Figure 4 shows the nitrogen adsorption/desorption isotherms for these modified carbon samples. The nitrogen adsorption/desorption results (Table 2; Fig. 4) show that the surface area is not much affected by the high temperature (C4-C at 723 K) and high-temperature steam treatments (C4-D at 723 K). However, the high-temperature steam treatment (C4-E at 823 K) and controlled air decomposition at 673 K (C4-F) and 773 K (C4-G) show slight decrease in the surface area. In the case of the controlled air decomposition carbon samples, the reduction in the surface area is due to the oxidation of a certain portion of the surface. The higher oxidation temperature further reduces the surface area as observed in the case of the carbon (C4-G) sample.

The BET curves for the temperature deposition of organics (n-hexane and toluene) on the microporous carbon are presented in Fig. 5. The deposition of n-hexane and toluene was carried out in order to see the effect of the aliphatic and aromatic hydrocarbons on the micropores and surface area, so

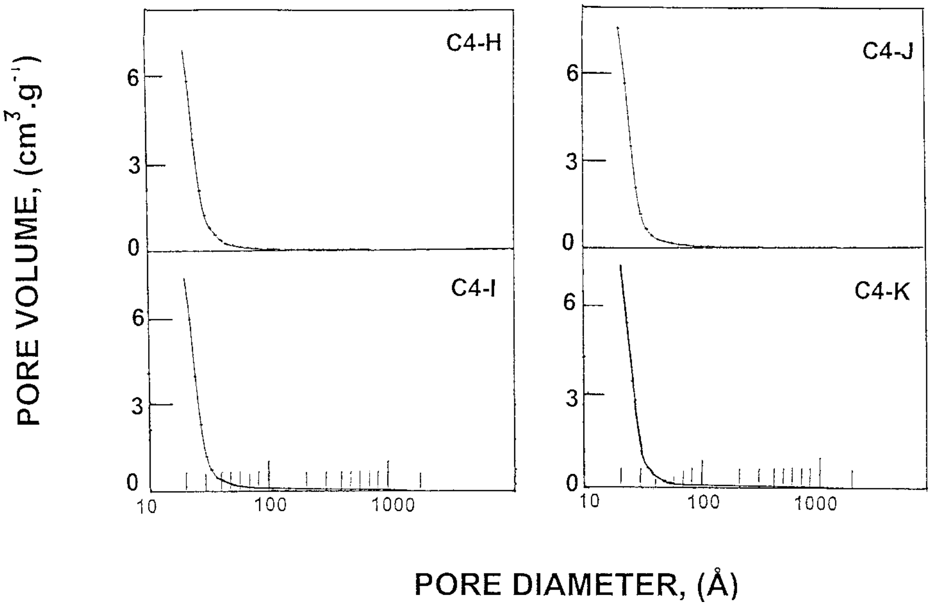

FIG. 10. The pore volume distributions (adsorption) for the carbon samples modified by high-temperature aliphatic and aromatic hydrocarbon treatments.

that this technique could be used to finely modify the pore system of the carbons. The BET data (Table 2) indicates that the type and amount of organic hydrocarbon and deposition temperature exhibits substantial influence on the surface area and micropore volume. The deposition of aliphatic hydrocarbon (*n*-hexane) shows a greater decrease in the micropore area and volume than that caused by the deposition of aromatic hydrocarbon (toluene). The high temperature hydrocarbon deposition shows an overall decrease in the surface area and micropore volume as compared to that seen with the fresh carbon sample.

The thermal, hydrothermal, acid–base, high-temperature controlled air, and hydrocarbon treatments exhibits significant influence on the pore size distribution of the macro-, meso-, and microporous carbon samples. The pore size distributions obtained by BET for the fresh and treated carbons are presented in Figs. 6–10. The hydrothermal, acid–base treatments of the macro- and mesoporous (C1, C2, C3) and microporous carbons show an increase in their average pore diameter (Table 2). The average pore diameter of these carbons increases with the treatment temperature. It seems that during the hydrothermal or acid–base treatments, the number of micropores are decreased due to collapse of micropore structures, thus increasing the number of macro-/mesopores. In the case of the microporous carbon (Figs. 9 and 10), the average pore diameter and total pore volume and micropore volume are reduced by the thermal, high-temperature steam, controlled air, and hydrocarbon deposition treatments.

In order to investigate the thermal decomposition pattern of the hydrothermally treated carbons, thermal gravimetric

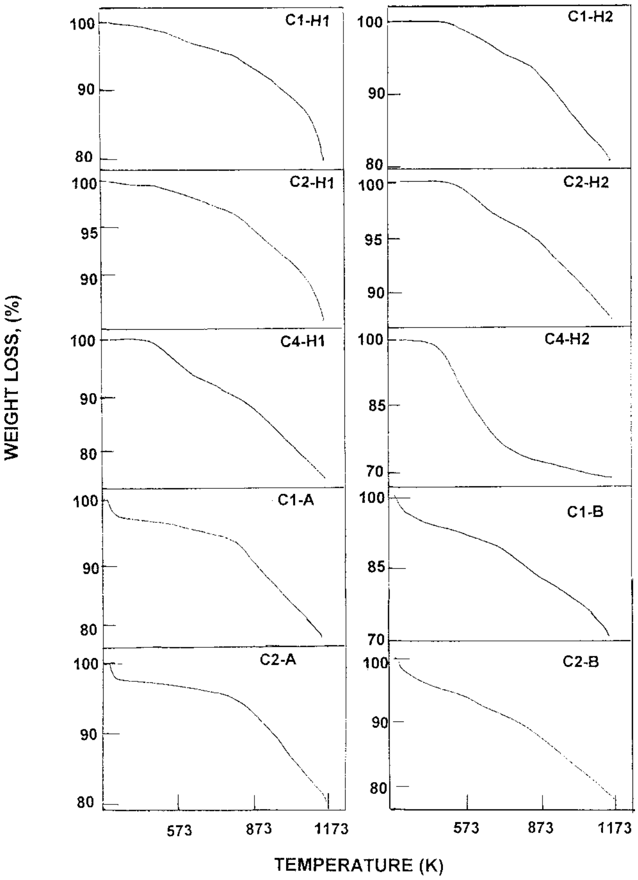

FIG. 11. Thermogravimetric curves for the hydrothermally modified carbon samples in neutral, acidic, and basic medium at 473 K (atmosphere: inert).

TABLE 3
Thermogravimetric Data of the Carbon Samples in Nitrogen

Sample	Weight loss (%)					Total weight loss (%)
Temperature range (K):	303–723	723–823	823–923	923–1023	1023–1173	
C1	9.8	1.6	2.9	6.2	10.9	31.4
C1–H1	4.4	1.1	2.3	3.0	11.0	21.0
C1–H2	4.0	1.5	2.5	5.0	6.0	19
C1–A	4.5	1.6	6.4	4.4	5.6	22.5
C1–B	11.8	4.2	3.2	4.0	7.8	30.0
C2	2.3	1.1	1.2	1.9	11.4	17.9
C2–H1	3.0	1.3	1.8	3.0	5.9	15.0
C2–H2	3.0	1.3	2.2	2.0	4.0	12.5
C2–A	5.0	0.9	2.1	5.2	6.8	20.0
C2–B	8.1	3.0	1.9	4.0	6.0	23.0
C4	3.2	1.8	2.3	3.2	4.1	14.6
C4–H1	8.0	4.0	3.0	5.0	5.0	25
C4–H2	24.0	2.0	2.0	1.5	1.0	30.5

analysis was performed in inert and oxidizing atmospheres. Figures 11 show the thermogravimetric curves for the decomposition of the hydrothermally treated carbon samples (prepared from mustard and almond oils and synthetic sources) in neutral, acidic, and basic medium at 473 K in nitrogen. It is interesting to note that the carbons prepared by different treatments show different decomposition modes, with the total decomposition in the presence of nitrogen being in the range 12–31%.

Thermogravimetric data obtained in different temperature ranges is presented in Table 3. The thermal stability of carbon samples prepared from mustard and almond oils increases with the hydrothermal treatment. The weight loss in the temperature decomposition steps (303–723, 723–823, 823–923, 923–1023, and 1023–1173 K) is different for the hydrothermally treated carbons. The weight loss is higher for the untreated carbons prepared from the mustard and olive oils than the hydrothermally treated carbons. The hydrothermal treatment reduces the total weight loss (at 1173 K) (Table 3) of the carbons prepared from mustard and almond oils which indicates the increase in the thermal stability of the carbons after the hydrothermal treatment. The high surface area carbon shows reverse trend. The high surface area carbon losses more weight at 1173 K after the hydrothermal treatment, indicating the decrease in its thermal stability. In the case of the microporous carbon (Table 3), the hydrothermal treatment decreases thermal stability of the material almost by twofold. However, the acid–base treatment decreases the thermal stability of carbon samples prepared from almond oil. The treated carbons exhibit different decomposition modes as compared with their respective parent sources.

The thermogravimetric decomposition curves of the treated macro-, meso-, and microporous carbons in an oxidizing atmosphere (air) are presented in Fig. 12. In an oxidizing atmosphere, the treated carbon samples show different decomposition modes compared to those observed in an inert atmosphere. The observed total decomposition of the carbons in the presence of air is 100% in the temperature range 843–1023 K.

Table 4 shows the thermogravimetric data obtained over different temperature ranges. In an oxidizing atmosphere, the hydrothermally and acid–base-treated treated macro-, meso-, and microporous carbon samples showed more weight loss below 923 K than seen with the respective

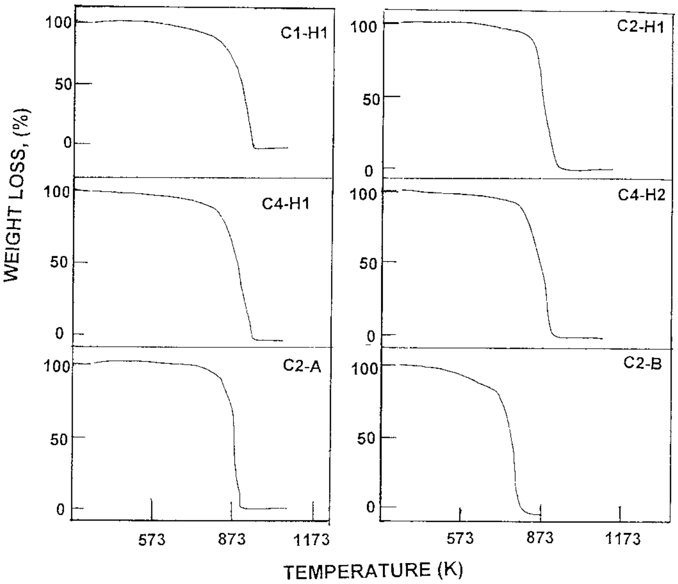

FIG. 12. Thermogravimetric curves for the hydrothermally modified carbon samples in neutral acidic and basic medium at 473 K (atmosphere: oxidizing).

TABLE 4
Thermogravimetric Data of the Carbon Samples in Air

Sample	Weight loss (%)				
Temperature range (K):	303–723	723–823	823–923	923–1023	Total weight loss (%)
C1	1.4	7.3	91.3		100
C1–H1	5.0	8.0	72.0	15.0	100
C2	0.5	2.3	34.5	62.8	100
C2–H1	3.0	9.5	74.5	13.0	100
C2–A	0.5	8.1	91.5		100
C4	1.4	2.2	30	66.4	100
C4–B	22.0	78.0			100
C4–H1	9.0	21.0	61.2	8.8	100

original untreated carbon. The acid-treated carbon prepared from the almond oil and the base-treated microporous carbon decomposes completely at lower temperatures than that of the parent carbon samples. The hydrothermally, acid–base-treated macro-, meso-, and microporous carbon samples showed lower thermal stability in air and their complete thermal degradation was observed at or below 1014 K.

Figure 13 shows the mid-FTIR spectra of the untreated and hydrothermally treated macro-, meso-, and microporous carbon samples. The macro-, meso-, and microporous carbon samples (C1–C4) show different spectrum in the lattice vibration region (1700–400 cm^{-1}). The carbons prepared from mustard oil (C1), almond oil (C2), and olive oil (C3) and the high surface area microporous carbon (C4) show the presence of phenolic O–H, aliphatic and aromatic C–H, aromatic ring, C–O as in phenols/ethers/esters, and conjugate aromatic C=O groups. For these carbons, the IR bands are assigned as 3300 cm^{-1}, phenolic O–H; 3000 cm^{-1}, aromatic C–H; 2950 cm^{-1}, aliphatic C–H; 1600 cm^{-1}, aromatic ring vibration or conjugate aromatic C=O; 1450 cm^{-1}, aliphatic C–H; 1380 cm^{-1}, aliphatic C–H; 1200 cm^{-1}, C–O as in phenols/ethers/esters; and 800 cm^{-1}, aromatic ring C–H groups. The FTIR analysis of the spectra indicates that the concentration and distribution of these groups are different over the carbon obtained from different sources. Changes in the lattice vibration region are more pronounced after the hydrothermal treatment at 398 and 473 K. Comparison of the FTIR spectra of the untreated (C1, C2) and hydrothermally treated (C1–H1, C1–H2, C2–H1, C2–H2) carbons indicate that the hydrothermal treatment degrades and causes certain changes in the carbon structure. The hydrothermal treatment affects the aromatic ring/conjugate aromatic C=O (1600 cm^{-1}), aliphatic C–H (1450, 1380 cm^{-1}), and aromatic C–H (800 cm^{-1}) groups.

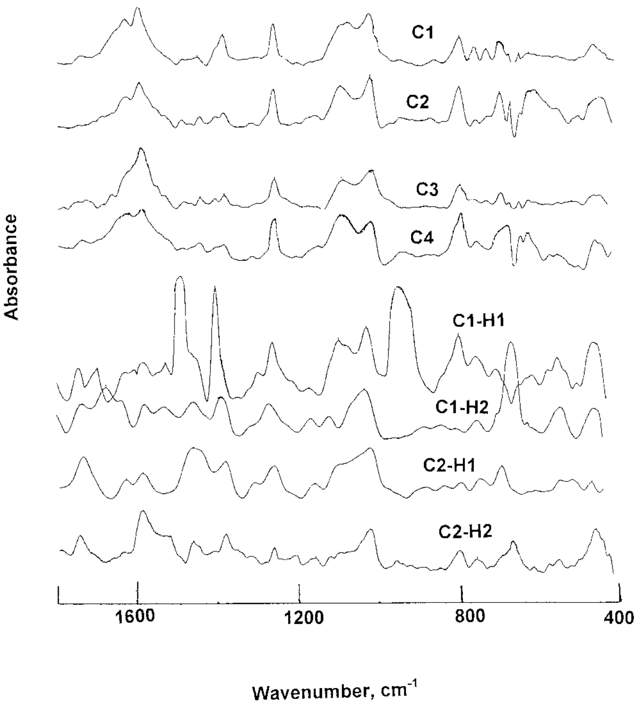

FIG. 13. Mid-FTIR spectra of fresh and hydrothermally modified carbon samples.

It is interesting to note that the FTIR spectra of the acid–base-treated macro-, meso-, and microporous carbon samples (Fig. 14) are totally different than those of the untreated and hydrothermally treated macro-, meso-, and microporous carbon samples. This shows that the acid–base treatment significantly affects the carbon structure and surface properties. The acid/base treatment of the carbons prepared from mustard and almond oils shows modification in the concentrations of phenolic O–H, aromatic C–H, and aliphatic C–H groups. However, the acid–base treatment given to the microporous carbon (C4) significantly reduces the concentrations of phenolic O–H, aliphatic C=O, and aromatic and aliphatic C–H groups.

Figure 15 shows the mid-FTIR spectra of the microporous carbon samples modified by controlled air decomposition and high-temperature aliphatic and aromatic hydrocarbons treatments. FTIR spectra show that the controlled air decomposition and high-temperature deposition of *n*-hexane and toluene strongly influence the structural properties of the microporous carbon. The controlled decomposition of the microporous carbon leads to the increase in the concentrations of aromatic C–H and aliphatic C–H and C–O (as in phenols/ethers) groups. The high-temperature deposition of *n*-hexane over the microporous carbon shows the increase in the concentration of aliphatic C–H group while the high-temperature deposition of toluene increases the concentration of aromatic ring and aromatic C–H as well as aliphatic C–H groups.

Comparison of the untreated and chemically treated carbon FTIR results indicates that the chemical treatments do change the concentrations of phenolic O–H, aromatic ring, C–O, C═O, aromatic C–H, and aliphatic C–H groups, etc., which strongly affects the structural and thermal stability as observed by the thermogravimetric analysis. The thermogravimetric analysis results indicated that the thermal stability of the macro-, meso-, and microporous carbons is significantly changed after the treatments. The results of the FTIR are in agreement with those of BET and thermogravimetric analysis, confirming that the treatments significantly affect the structural, surface, and sorption properties of the macro-, meso-, and microporous carbon samples.

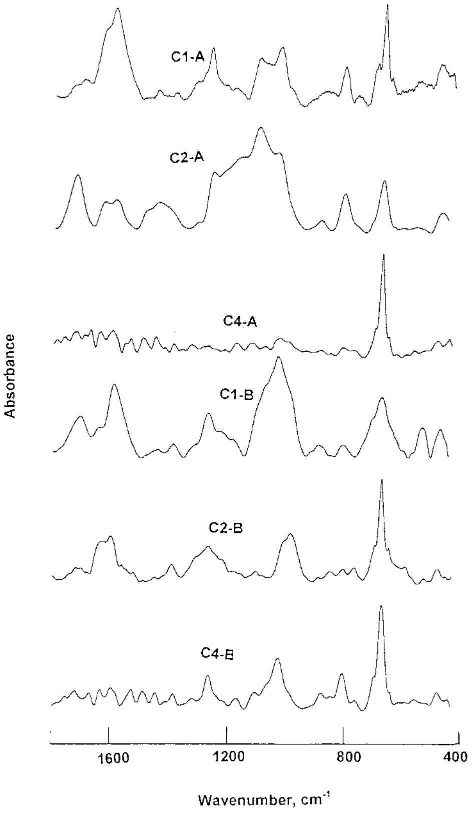

FIG. 14. Mid-FTIR spectra of the hydrothermally modified carbon samples in acidic and basic media at 473 K.

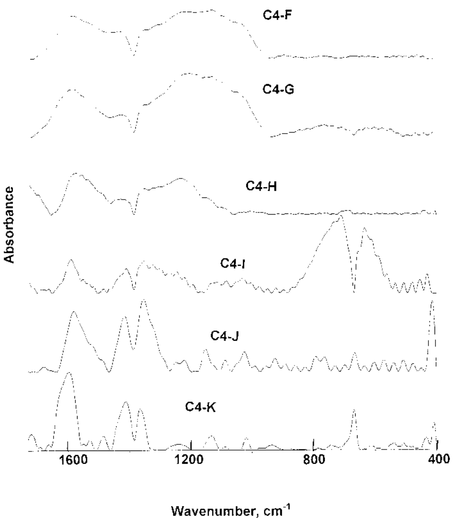

FIG. 15. Mid-FTIR spectra of the carbon samples modified by controlled decomposition and high-temperature aliphatic and aromatic hydrocarbon treatments.

CONCLUSIONS

The thermally, hydrothermally, acid–base, controlled high-temperature air and hydrocarbon-treated carbons (exhibiting low and high surface areas) obtained from natural and synthetic sources, show different surface properties, thermal stability (in nitrogen and air), and structure. The changes in the thermal/structural stability, surface area, and pore size distribution vary with the type of sample treatment. Comparison of the BET, thermogravimetric, and FTIR results indicates that the surface, thermal, and structural properties of the macro-, meso-, and microporous carbons are strongly affected by the hydrothermal and high-temperature acid–base treatments. In the case of the microporous carbon, the controlled air decomposition and hydrocarbon deposition treatments reduce the surface area and modify the structural properties. The thermal, hydrothermal, acid–base, controlled air, and hydrocarbon treatment methods could be easily applied for modifying the properties of carbon materials.

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